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Organometallic sulfur complexes

III *. Reactions of organoiron sulfanes, $(\mu\text{-}S_x)[FeCp(CO)_2]_2$ $(x = 3, 4)$ and the synthesis of cyclopentadienyldicarbonyliron *S*-bonded monothiocarboxylates, $FeCp(CO)_2(SCOR)$

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Abstract

The reaction of organoiron sulfanes, $(\mu\text{-}S_x)[FeCp(CO)_2]_2$ $(x = 3, 4)$ with $LiBEt_3H$ at −97 °C gives the anionic species $[Cp(CO)_2FeS_x]^-$ $(x = 1, 2$ or $3)$. The solution containing the anionic species reacts with acid chlorides RCOCl to give $[FeCp(CO)_2(SCOR)]$, which contain the *S*-bonded monothiocarboxylate ligand. The same products and $[FeCp(CO)_2Cl]$ were obtained from the reaction of $(\mu\text{-}S_x)[FeCp(CO)_2]_2$ $(x = 3,4)$ and RCOCl at room temperature.

Introduction

The synthesis and molecular structure of the binuclear tri- and tetra-sulfur bridged organoiron complexes $(\mu\text{-}S_x)[Fe(\eta^5\text{-}C_5H_5)(CO)_2]_2$ $(x = 3, 4)$, which may be discribed as organoiron sulfanes were reported recently [2]. These complexes are fairly stable as solids, but in solution they are sensitive to oxygen and light. The reactivities of these organoiron sulfanes are of interest, since they contain potentially reactive S–S–S bonds and the sulfur atoms of the bridged S_x $(x = 3, 4)$ ligands are potential electron donors. The PPh_3 desulfurizes the organoiron sulfane [2] in the same way as it does organic trisulfides [3], as shown in eq. 1 and 2.

$$\text{Fp–S–S–S–Fp} + P(C_6H_5)_3 \rightarrow \text{Fp–S–S–Fp} + SP(C_6H_5)_3 \qquad (1)$$

$(\text{Fp} = FeCp(CO)_2)$

$$\text{R–S–S–S–R} + P(C_6H_5)_3 \rightarrow \text{R–S–S–R} + SP(C_6H_5)_3 \qquad (2)$$

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The organoiron disulfane produced from reaction 1 was rapidly desulfurized on adding one equivalent of $P(C_6H_5)_3$ to give the iron monosulfane. The latter also reacts with $P(C_6H_5)_3$ to give Fp_2 and $SP(C_6H_5)_3$. Thermolysis (refluxing in toluene for 6–8 h) of the organoiron sulfanes gives an almost quantitative yield of the well known iron tetramer, $[Fe(C_5H_5)S]_4$ [2].

It is known that the S–S bond of organic disulfide or trisulfide is reactive toward diverse nucleophilic, electrophilic and radical reagents [4]. In this study we have investigated the reactions of the organoiron trisulfane and tetrasulfane with acid chlorides, as electrophiles. The formation of organoiron sulfide, FpS^-, from the reaction of either the trisulfane or tetrasulfane with $LiEt_3BH$ and the reaction of FpS^- with acid chlorides was also studied. In the course of this investigations we obtained a series of organoiron *S*-bonded monothiocarboxylates, $Fe(\eta^5\text{-}C_5H_5)(CO)_2\text{-}SCOR$. The synthesis and characterization of these complexes are reported in this paper.

Experimental

All reactions were performed under nitrogen with Schlenk glassware [5]. Solvents were dried and purified as previously described [2]. Acid chlorides were commercial samples. An 1.0 *M* THF solution of $LiEt_3BH$ was used as purchased. The organoiron sulfane complexes, $(\mu\text{-}S_x)[Fe(\eta^5\text{-}C_5H_5)(CO)_2]_2$ ($x = 3, 4$) were prepared as described in the literature [2]. ^1H NMR spectra were recorded on Bruker WP 80 SY spectrometer with TMS as internal standard. Mass spectra were recorded on VG analytical 7070E spectrometer (EI low resolution). Infrared spectra were recorded on Pye-Unicam SP3-100 spectrophotometer, KBr cells being used for solutions.

Table 1

Yields, melting points and analytical data of organoiron *S*-bonded monothiocarboxylates, $FeCp(CO)_2\text{-}SCOR$

$FeCp(CO)_2SCOR$	R	Yield (%) (Procedure)	M.p. (°C)	Analysis (Found (calc)(%))		
				C	H	S
Compound I	CH_3	25 (I)	59–60	42.87	3.42	12.54
		40 (II)		(42.86)	(3.17)	(12.70)
Compound II	C_6H_5	30 (I)	125–127	53.40	3.41	10.03
		50 (II)		(53.50)	(3.18)	(10.19)
Compound III	$2\text{-}C_4H_3O$	35 (I)	113–115	47.13	2.76	10.67
	2-furyl	55 (II)		(47.37)	(2.63)	(10.53)
Compound IV	$3\text{-}MeOC_6H_4$	25 (I)	72–73	52.13	3.61	9.06
		30 (II)		(52.33)	(3.49)	(9.30)
Compound V	$C(CH_3)_3$	28 (I)	81–82	48.73	4.59	10.67
		36 (II)		(48.98)	(4.76)	(10.88)
Compound VI	$4\text{-}O_2NC_6H_4$	48 (I)	130–131	46.81	2.44	8.99
		56 (II)		(46.80)	(2.51)	(8.91)
				N, 3.99 (3.98)		
Compound VII	$1\text{-}C_{10}H_7$	34 (I)	147–149	59.12	3.51	8.51
	1-naphthyl	40 (II)		(59.34)	(3.30)	(8.79)
Compound VIII	FpSCO–COSFp	20 (I)	97–98	40.36	2.21	13.69
		30 (II)		(40.51)	(2.11)	(13.50)

Table 2. IR and ^1H NMR spectra of $FeCp(CO)_2SCOR$ compounds I–VIII

Compound	IR (CH_2Cl_2) [a] (cm^{-1})	^1H NMR ($CDCl_3$) [b] (δ, ppm)
I R = CH_3	3050 vw, 2910 m 2850 m, *2040 vs* *1988 vs* (ν(C≡O)), *1625 s* ν(C=O), 1422 w, 1348 w, 1112 s, 1065 m 946 m, 844 m	2.40 (s, 3H, CH_3) 4.98 (s, 5H, C_5H_5)
II R = C_6H_5	3047 vw, 3023 vw *2034 vs*, *1990 vs* (ν(C≡O)) *1596 s* (ν(C=O)), 1572 s, 1420 w, 1200 s, 1174 s, 1079 w, 1005 m, 918 s, 845 s	5.05 (s, 5H, C_5H_5) 7.31–7.43 (m, 3H, *m*-, *p*-Ar*H*) 8.06–8.2 (m, 2H, O–Ar*H*)
III R = 2-C_4H_3O (2-furyl)	3050 vw, *2035 vs* *1995 vs* (ν(C≡O)), *1590 s* (ν(C=O)), 1555 m, 1466 m, 1382 w, 1225 m, 1015 m, 956 m, 860 s	5.07 (s, 5H, C_5H_5) 6.43 (m, 1H, C(4)-*H*) 7.08 (d, 1H, C(3)-*H*) 7.49 (s(br), 1H, C(5)-*H*)
IV R = 3-$MeOC_6H_4$	3040 vw, 2910 w, *2030 vs*, *1989 vs* (ν(C≡O)) *1578 s* (ν(C=O)), 1460 w, 1245 w, 1151 m, 1045 m, 947 w, 843 m,	3.83 (s, 3H, OCH_3) 5.06 (s, 5H, C_5H_5) 6.95–7.4 (m, 2H, Ar*H*) 7.6–7.82 (m, 2H, Ar*H*)
V R = $C(CH_3)_3$	3050 vw, 2920 m *2028 vs*, *1987 vs* (ν(C≡O)) *1610 s* (ν(C=O)) 1468 w, 1375 w 1041 w, 952 m 847 m,	1.25 (s, 9H, $C(CH_3)_3$) 4.97 (s, 5H, C_5H_5)
VI R = 4-$O_2NC_6H_4$	3050 w, 2920 w *2040 vs*, *1998 vs* (ν(C≡O)) *1591 s* (ν(C=O)) 1520 m, 1425 w 1352 s, 1321 m 1191 m, 1173 m 1113 w, 1019 w 931 s, 868 m, 855 s	5.09 (s, 5H, C_5H_5) 8.21 (s, 4H, Ar*H*)
VII R = 1-$C_{10}H_7$ (1-naphthyl)	3047 vw, 3010 vw *2035 vs*, *1990 vs* (ν(C≡O)) *1605 s* (ν(C=O)) 1225 m, 1173 m 1081 m, 1058 m 925 m, 847 m	5.08 (5H, C_5H_5) 7.3–8.34 (m's, 7H, Ar*H*)
VIII FpSCO–COSFp	3040 w, *2038 vs*, *1995 vs*, (ν(C≡O)) *1609 s*, (ν(C=O)) 1125 m, 1093 m 912 w, 852 m	5.05 (s, H'S, C_5H_5)

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[a] vs, very strong; s, strong; m, medium; w, weak; vw, very weak. [b] s, singlet; d, doublet; m, multiplet, br, broad.

Table 3

Low resolution mass spectra of $FeCp(CO)_2SCOR$ compounds I–VIII

Compounds	m/e (rel. intensity)
I R = CH_3	252 (M^+, 5), 224 (M^+ – CO, 17) 196 (M^+ – 2CO, 42), 153 ($CpFeS^+$, 45) 121 ($CpFe^+$, 28), 65 (Cp^+, 29) 56 (Fe^+, 20), 43 (CH_3CO^+, 87) 32 (S^+, 72)
II R = C_6H_5	258 (M^+ – 2CO, 6), 121 ($CpFe^+$, 31) 105 ($PhCO^+$, 91), 77 (Ph^+, 62) 65 (Cp^+, 12), 56 (Fe^+, 32)
III R = 2-C_4H_3O (2-furyl)	276 (M^+ – CO, 12), 248 (M^+ – 2CO, 66) 177 ($Cp(CO)_2Fe^+$, 5), 153 ($CpFeS^+$, 7) 121 ($CpFe^+$, 72), 95 ($C_4H_3OCO^+$, 100) 65 (Cp^+, 8), 56 (Fe^+, 29)
IV R = 3-$MeOC_6H_4$	288 (M^+ – 2CO, 9), 177 ($Cp(CO)_2Fe^+$, 6) 153 ($CpFeS^+$, 8), 135 ($MeOPhCO^+$, 94) 121 ($CpFe^+$, 20), 107 ($MeOph^+$, 22) 77 (Ph^+, 21), 65 (Cp^+, 12) 56 (Fe^+, 15)
V R = $C(CH_3)_3$	294 (M^+, 7), 266 (M^+ – CO, 16) 238 (M^+ – 2CO, 100), 177 ($Cp(CO)_2Fe^+$, 22) 153 ($CpFeS^+$, 19) 149 $CpCOFe^+$, 13) 121 ($CpFe^+$, 76) 85 ($(CH_3)_3CCO^+$, 20) 32 (S^+, 69)
VI R = 4-$O_2NC_6H_4$	359 (M^+, 6), 331 (M^+ – CO, 8) 303 (M^+ – 2CO, 40) 150 (O_2NPhCO^+, 16) 121 ($CpFe^+$, 13) 65 (Cp^+, 54), 32 (S^+, 87)
VII R = 1-$C_{10}H_7$ (1-naphthyl)	364 (M^+, 7), 336 (M^+ – CO, 6) 308 (M^+ – 2CO, 28) 155 ($C_{10}H_7CO^+$, 100) 149 ($Cp(CO)Fe^+$, 10), 127 ($C_{10}H_7^+$, 100) 121 ($CpFe^+$, 13), 65 (Cp^+, 31) 56 (Fe^+, 16), 32 (S^+, 57)
VIII FpSCO–COSFp	209 ($Cp(CO)_2FeS^+$, 6) 181 ($Cp(CO)FeS^+$, 4) 153 ($CpFeS^+$, 13) 121 ($CpFe^+$, 16)

Elemental analyses (Table 1) were performed by M.H.W. Laboratories. Phoenix, Arizona 85018. Spectral data are reported in Tables 2 and 3.

General procedures

I. Formation of Li[FeCp(CO)$_2$S] (Cp = η^5-C_5H_5) and its reactions with acid chlorides. A reddish-brown solution of 2.0 mmol of (μ-S_x)$[FeCp(CO)_2]_2$ (x = 3 or

4) in 50 cm^3 of THF was cooled to $-97\,^\circ$C (methylene chloride slush bath) and 4 cm^3 of 1.0 *M* THF solution of $LiEt_3BH$ (4 mmol) was added slowly from a syringe. To the dark greenish-brown solution formed 4 mmol of the acid chloride RCOCl (or 2 mmol of the diacid chloride ClCO–COCl) in THF (10 cm^3) was added slowly at $-97\,^\circ$C. The brown mixture was allowed to warm to room temperature with stirring, then a sample was examined by TLC (SiO_2, CH_2Cl_2), which showed the formation of single orange-yellow product. The mixture was dried under vacuum and the solid residue was extracted with CH_2Cl_2 and the extract filtered. The extract was subjected to column chromatography (70–230 mesh silica gel, CH_2Cl_2; CH_2Cl_2/CH_3OH 100/5, for compound VIII), the recovered product recrystallized from CH_2Cl_2/n-hexane to give orange to reddish-orange crystals of the organoiron *S*-bonded monothiocarboxylates, [FeCp(CO)$_2$(SCOR)] or the dimonothiooxalate [Cp(CO)$_2$FeSCO–COSFe(CO)$_2$Cp].

II. Reactions between the organoiron sulfanes (μ-S$_x$)[FeCp(CO)$_2$]$_2$ (x = 3 or 4) and acid chlorides. To a reddish brown solution of 0.9 g (2.0 mmol) of (μ-S$_3$)-[FeCp(CO)$_2$]$_2$ in diethyl ether (50 cm^3) a solution of acid chloride (2–2.5 mmol for a monoacid chloride and 0.5–1.0 mmol for ClCO–COCl) in diethyl ether (10 cm^3) was added slowly at room temperature. The colour changed to orange within 15–30 min. After 1–2 h a sample of the mixture was examined by TLC (Silica, CH_2Cl_2), which showed the formation of an orange-yellow product along with a red product with a lower R_f value. These were separated by column chromatography (70–230 mesh silica gel, CH_2Cl_2, CH_2Cl_2/CH_3OH, 100/5 for compound VIII). The orange product eluted first was recrystallized from CH_2Cl_2/n-hexane to orange to reddish-orange crystals of [FeCp(CO)$_2$(SCOR)]. The red product was obtained as red crystals (yield ~ 10%, based on RCOCl) from a mixture of CH_2Cl_2/petroleum ether (60–80) and identified as [FeCp(CO)$_2$Cl] [6] (m.p. 87–88 °C). ^{1}H NMR ($CDCl_3$); singlet at δ 5.05 ppm. IR (CH_2Cl_2) ν(CO) 2050s and 2004s cm^{-1}. Analysis Found: C, 39.3; H, 2.53; Cl, 16.57. $C_7H_5FeO_2Cl$ calcd.: C, 39.6; H, 2.35; Cl, 16.7%.

The yields, melting points and analytical data of the organoiron *S*-bonded monothiocarboxylates FeCp(CO)$_2$SCOR prepared by the above procedures are shown in Table 1.

Results and discussion

Lithium triethylborohydride is known to cleave organic disulfides to form lithium organic sulfide [7], and dinuclear metal carbonyls to give metal-centered anions [8]. It has been reported that the reduction of μ-dithio bis(tricarbonyliron), [(μ-S$_2$)Fe$_2$(CO)$_6$], to the sulfur-centered dianion, [(μ-S$_2$)Fe$_2$(CO)$_6$]$^{2-}$ is best achieved by use of lithium triethylborohydride. A study of the reactions of the dianion with diverse organic and inorganic electrophiles revealed a marked similarity between the reactions of (μ-S$_2$)Fe$_2$(CO)$_6$ and those of organic disulfides [9]. In a recent report the monoanions and dianion obtained from the reduction of (μ-S$_2$)Fe(CO)$_6$ with PhLi or *p*-tolylmagnesium bromide were used as nucleophiles and treated with acid chlorides, to give the iron carbonyl derivatives of the *S*-bonded monothiocarboxylic acids RCOSH [10]. In our work when the reddish-brown solution of (μ-S$_3$)[FeCp(CO)$_2$]$_2$ in THF was reduced with two molar equivalents of $LiBEt_3H$ at $-97\,^\circ$C a greenish-brown solution was obtained, and addition of two equivalents of acid chloride (or one equivalent of the diacid chloride, ClCO–COCl) to the solution at −97 °C changed the colour to brown and gave the organoiron *S*-bonded thiocarboxylates, $FeCp(CO)_2SCOR$, which were isolated in moderate to low yields. The structural relation between the reactants in the solution and the isolated products suggested that the intermediate formed in the reduction and treated with the acid chloride to give the final product was the organoiron sulfide FpS_x^- ($x = 1$, 2), as shown in equations 3–5.

$$\text{Fp–S–S–S–Fp} + 2\text{LiBEt}_3\text{H} \rightarrow \text{FpS}^-\text{Li}^+ + \text{FpS}_2^-\text{Li}^+ + 2\text{BEt}_3 + \text{H}_2 \quad (3)$$

$$\text{FpS}^-\text{Li}^+ + \text{RCOCl} \rightarrow \text{FpSCOR} + \text{LiCl} \quad (4)$$

$$\text{FpS}_2^-\text{Li}^+ + \text{RCOCl} \rightarrow \text{FpS}_2\text{COR} + \text{LiCl} \quad (5)$$

These organoiron sulfides are expected to be reactive toward many organic and inorganic electrophiles, as are organic sulfide RS^- [11]. Since only one single organoiron *S*-bonded monothiocarboxylate product, $FeCp(CO)_2SCOR$, was obtained from the reaction at room temperature, the expected compound, FpS_2COR, shown in eq. 5 appeared to be unstable, and may decompose into more stable FpSCOR or to other insoluble unidentified compounds. Addition of four molar equivalents of $LiBEt_3H$ to the organoiron sulfane produces a dark green solution, and reaction of the resulting green organoiron sulfide solution with two equivalents of the acid chloride did not appreciably improve the yield of FpSCOR. The dark green solution is assumed to contain only FpS^- species, as in eq. 6:

$$\text{Fp–S}_3\text{–Fp} + 4\text{LiBEt}_3\text{H} \rightarrow 2\text{FpS}^-\text{Li}^+ + \text{Li}_2\text{S} \quad (6)$$

The above results rule out the formation of the iron-centered anion, Fp^-, in the lithium triethylborohydride reduction of the organoiron sulfane. The reduction of the organoiron tetrasulfane, $(\mu\text{-S}_4)[FeCp(CO)_2]_2$ with either two, four or six equivalents of the hydride reagent and reaction of the formed organoiron sulfide species FpS_x^- ($x = 3$, 2, 1) with the acid chloride gives the single organoiron *S*-bonded monothiocarboxylate, FpSCOR. Addition of $LiBEt_3H$ to the iron sulfane solution at room temperature produced a black solution; when this was treated with acid chloride no organoiron monothiocarboxylate complex was obtained, but instead a dark-brown or black precipitate of unknown composition.

The reactions of organoiron trisulfane or tetrasulfane with the acid chlorides in 1/1 molar ratio readily proceed to completion at room temperature within 1–2 h, with colour change from reddish-brown to brownish-orange. The reactions gave two products, the organoiron *S*-bonded monothiocarboxylate, $FeCp(CO)_2SCOR$, and the organoiron chloride, $FeCp(CO)_2Cl$. The two products were separated by column chromatography. The reaction of the organoiron sulfane with acid chloride can be represented as in eq. 7 and 8, or possibly 9–11.

$$\text{Fp–S}_3\text{–Fp} + \text{RCOCl} \rightarrow \text{FpSCOR} + \text{FpS}_2\text{Cl} \quad (7)$$

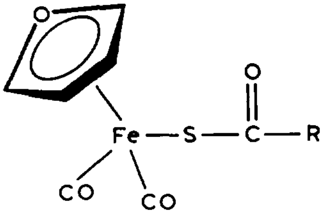
$$\text{FpS}_2\text{Cl} \rightarrow \text{FpCl} + \tfrac{1}{4}\text{S}_8 \quad (8)$$

$$\text{Fp–S}_3\text{–Fp} + \text{RCOCl} \rightarrow \text{FeS}_2\text{COR} + \text{FpSCl} \quad (9)$$

$$\text{FpSCl} \rightarrow \text{FpCl} + \text{S} \quad (10)$$

$$\text{Fp–S}_3\text{–Fp} \rightarrow \text{RCOCl} \rightarrow \text{FpS}_3\text{COR} + \text{FpCl} \quad (11)$$

The most stable products, FpSCOR and FpCl are isolated, other products presumably being unstable.



R	Compound
CH_3	I
C_6H_5	II
$2\text{-}C_4H_3O$ (2-furyl)	III
$3\text{-}MeOC_6H_4$	IV
$C(CH_3)_3$	V
$4\text{-}O_2NC_6H_4$	VI
$1\text{-}C_{10}H_7$ (1-naphthyl)	VII

Fig. 1.

The reactivity of organoiron sulfanes toward acid chlorides as electrophiles is attributed to the reactivity of the sulfur atom as electron donor in the bridging S_x ($x = 3, 4$) group. Similar reactions were reported for the cubane iron-sulfide cluster [12,13], as shown in eq. 12, and for the coordinated dithiobis(dialkylphosphine) in the compound $L_nMnR_2P\text{–}S_2\text{–}PR_2MnL_n$; the latter complex reacts readily with RCOCl to give L_nMnR_2PSCOR in high yield [14].

The new organoiron *S*-bonded monothiocarboxylates, compounds I–VIII obtained in the present work are stable as solids, they are only slightly sensitive to oxygen in solution except for compound VIII, FpSCO–COSFp, which is more sensitive. The structural formula of $FeCp(CO)_2SCOR$ is shown in Fig. 1. Infrared and ^1H NMR spectral data are shown in Table 2.

The proton NMR spectra of each $FeCp(CO)_2SCOR$, shows the singlet peak due to Cp protons in the range 4.97–5.08 ppm typical of $[FeCp(CO)_2X]$ (X = Cl, Br) [15] and the peaks due to the R-protons with a pattern similar to that observed for the corresponding RCOCl compounds [16] but shifted slightly (0.15–0.25 ppm) up field.

The infrared spectra of the $[FeCp(CO)_2(SCOR)]$ compounds each show two strong carbonyl bands in the ranges 2040–2028 and 1995–1987 cm^{-1}, as observed in $FeCp(CO)_2X$ (X = Cl, Br) [15]. The monothiocarboxylate ligand can exhibit monodentate (*S*-bonded or *O*-bonded) or bidentate (chelating or bridging) coordination behavior [17]. The positions of $\nu(\mathrm{C}\overset{\cdots}{-}2\mathrm{O})$ and $\nu(\mathrm{C}\overset{\cdots}{-}2\mathrm{S})$ bands can be used to determine which of these coordination modes are present in a series of transition metal-thiobenzoate complexes [17,18]. In complexes, I–VIII, the fact that the ν(CO) bands are in the range 1625–1578 cm^{-1} and the $\nu(\mathrm{C}\overset{\cdots}{-}\mathrm{S})$ bands in the range 912–956 cm^{-1} indicates that the compounds contain the *S*-bonded monothiocarboxylate ligand [18].

The mass spectral results for compounds I–VIII are listed in Table 3. Some compounds show the molecular ion, M^+, but the others give as the highest mass ion $[M^+ - CO]$ or $[M^+ - 2CO]$. The most abundant ion for most compounds was RCO^+. To our knowledge, the only previously known organoiron *S*-bonded monothiocarboxylate, $FeCp(CO)_2SCOR$ (R = Ph), was prepared by King in 1963 [19] from the reaction of $[FeCp(CO)_2]_2$ with monothiobenzoic acid, and King also showed that treatment of monothioacids, RCOSH, with metal carbonyls gives a complex mixture resulting from complete loss of Cp and CO ligands. The compounds $(Me_3P)_2(CO)_3MSCOCH_3$ (M = Mn, Re) were prepared from the reaction of MeCOCl and $(Me_3P)_2(CO)_3MSH$ [20]. This reaction is similar to that of organic thiols with acid chlorides to form thioesters. The reactions we have described here may provide convenient and general routes to new cyclopentadienyl dicarbonyliron derivatives of monothiocarboxylic acids, RCOSH. The reactions of these compounds are now under investigation.

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